

Water-Soluble Phosphorus in Inorganic Liquid Fertilizer

1. Scope:

To provide a standardized procedure for the gravimetric analysis of water-soluble (available) phosphorus in inorganic liquid fertilizer using the quinolinium molybdophosphate (QMP) method.

2. Principle:

Samples are digested with 50% nitric acid and the available phosphorus is precipitated with Quimociac reagent. The resulting precipitate is filtered, washed, dried, and weighed to calculate the amount of available phosphorus (as P_2O_5) present in the sample. Samples are prepared according to Sample Preparation, Storage, and Disposal (RA-SP-SMPL-PREP).

3. Safety:

- 3.1. All laboratory safety rules for chemical handling, sample preparation, and analysis shall be followed. Read the SDS for all materials before use.
- 3.2. Nitric acid is highly corrosive. Preparation of the Quimociac reagent and the 50% nitric acid solution shall be done in a fume hood using appropriate personal protective equipment (gloves, eye protection, etc.)

4. Definitions:

QMP = quinolinium molybdophosphate = $(C_9H_7N)_3H_3PO_4 \cdot 12MoO_3$

5. Equipment (equivalents are acceptable):

- 5.1. Analytical balance capable of weighing to 0.0001g
- 5.2. Oven capable of $250^\circ C \pm 25^\circ C$
- 5.3. Hot plate
- 5.4. Volumetric flat bottom boiling flask – 250mL
- 5.5. Erlenmeyer flask – 500mL
- 5.6. Vacuum filter flask with adapter – 2L
- 5.7. Gooch crucibles
- 5.8. Glass fiber filters – 2.4cm circles (Whatman 934-AH)
- 5.9. Glass fiber filters – 11cm circles (Whatman 934-AH)
- 5.10. Boiling chips (micro granules)
- 5.11. Desiccator

6. Reagents and Supplies (equivalents are acceptable):

- 6.1. Nitric acid, concentrated (Fisher cat# A509-P212)
- 6.2. Sodium molybdate dihydrate (Fisher cat# S336-3)
- 6.3. Citric acid (VWR cat# BDH9228)
- 6.4. Synthetic quinoline (Acros Organics cat# 221141000 or Sigma Aldrich cat# 241571)
- 6.5. Acetone (Fisher cat# A949-4)

7. Preparation of Reagents:

- 7.1. Prepare the 50% nitric acid by mixing 1000mL with 1000mL water.
- 7.2. Prepare the Quimociac reagent:
 - 7.2.1. Dissolve 70g sodium molybdate dihydrate in 150mL water.
 - 7.2.2. In a 1L volumetric flask, dissolve 60g citric acid in a mixture of 85mL concentrated nitric acid and 150mL water. Allow to cool.
 - 7.2.3. Gradually add the sodium molybdate solution to the citric acid solution while stirring.
 - 7.2.4. Dissolve 5mL synthetic quinoline in a mixture of 35mL concentrated nitric acid and 100mL water.
 - 7.2.5. Gradually add the quinoline solution to the molybdate-citric acid solution. Mix and let stand for 24 hours.
 - 7.2.6. Filter solution through an 11cm glass fiber filter.
 - 7.2.7. Add 280mL acetone and fill to the mark with water.

8. Analysis

- 8.1. Perform the daily balance verification.
- 8.2. Weigh ~1g sample into a 250mL boiling flask (may need up to 2g of sample if guarantee is <1%). Record weight to nearest 0.0001g.
- 8.3. Add ~100mL water.
- 8.4. Add 10mL 50% nitric acid solution, boiling chips, and place on a hot plate in a fume hood. Gently boil for ~10 minutes. Do not let the flask boil to dryness.
- 8.5. Remove the flask from the hot plate, fill the flask almost to the 250mL mark with water and cool to room temperature.
- 8.6. Once cool, fill to the mark with water, tightly stopper, and mix. Allow particulates to settle overnight.

- 8.7. Pipette into a 500mL Erlenmeyer flask a suitable aliquot of the clear supernatant to form ~0.3g precipitate. If the guarantee is $\leq 5\%$, aliquot 50mL. If the guarantee is 5-10%, aliquot 25mL. If the guarantee is $>10\%$, aliquot 15mL.
- 8.8. Add water to bring the total volume to ~100mL.
- 8.9. Heat on a hot plate set to 350°C for ~15 minutes. Remove from the hot plate, swirl the solution, and add 50mL of Quimociac reagent.
- 8.10. Swirl the solution again, return to the hot plate, and gently boil the precipitate solution for 1 minute.
- 8.11. Remove from the hot plate and allow to cool to room temperature. Carefully swirl the solution 3-4 times during the cooling process.
- 8.12. Weigh a Gooch crucible fitted with a glass fiber filter. Record the weight to the nearest 0.0001g.
- 8.13. Using the vacuum flask and vacuum, filter the precipitate into the crucible.
- 8.14. Wash the precipitate with five 5mL portions of water, allowing each portion to drain thoroughly before adding the next.
- 8.15. Dry the crucible for 45 minutes in an oven preheated to 250°C.
- 8.16. Cool in a desiccator to room temperature.
- 8.17. Weigh the crucible and record weight to nearest 0.0001g. Subtract the weight of the crucible and filter from step 8.12 to determine the weight of the precipitate.
- 8.18. If the weight of the precipitate is greater than 1.0g, repeat steps 8.7 – 8.17 using a smaller aliquot of clear supernatant.

9. QA/QC:

- 9.1. A laboratory control sample (LCS) shall be run with each set. An acceptable LCS is a Magruder check sample with the reported mean and standard deviation for the direct available phosphate, gravimetric Quimociac (Magruder method code 041.1). An acceptable recovery is ± 2 standard deviations.
- 9.2. The reporting limit (RL) is 0.05%.

10. Calculations:

Calculate percent available phosphoric acid (P_2O_5):

$$\% P_2O_5 = \frac{W * D * 0.03207 * 100}{S}$$

Where:

W = Weight (g) of precipitate from step 8.17

D = Dilution factor = 250mL/aliquot

S = Sample weight (g)

0.03207 = Gravimetric factor derived from

Molecular weight of P_2O_5 = 141.94

Molecular weight of QMP = 2212.71

$$\frac{P_2O_5}{2QMP} = \frac{141.94}{2 * 2212.71} = 0.03207$$

11. References:

AOAC International Official Methods of Analysis, Method 962.03 (chapter 2.3.07), 17th edition, 2000.

USDA Food Safety and Inspection Service, Chemistry Laboratory Guidebook, Method 3.009, June 1987.

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